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SUPPLEMENTAL PLATFORM, POSTER AND BREAKOUT SESSION ABSTRACTS

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PIEZOELECTRIC MICROSENSORS: CURRENT AND FUTURE PERSPECTIVES

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Solid-state microsensor devices based on the modulation of piezoelectrically generated acoustic wave properties have demonstrated great potential as highly sensitive alternatives to more traditional electrochemical and spectrometric sensors. Piezoelectric microsensors fall into three general categories namely, bulk acoustic wave (BAW) devices, surface acoustic wave (SAW) devices and acoustic plate mode (APM) devices.

Bulk acoustic wave (BAW) devices have enjoyed considerable attention since Sauerbrey (1) first described the influence of surface mass changes on the resonant frequency of piezoelectric crystals in 1959. Since then they have been used in diverse applications including film thickness measurement, aerosol particle measurement, gas detection, and biosensors.

Surface acoustic wave (SAW) microsensor applications began to emerge in the late 1970's. Most work has focussed on using these devices for chemical vapor detection and probing the mechanical properties of thin polymeric films. SAW devices are more attractive than BAW devices for some applications owing to the significantly higher sensitivity afforded by their higher operating frequencies. One shortcoming of traditional Rayleigh wave SAW devices has been their unsuitability to liquid phase monitoring resulting from compressional wave losses into the liquid. Heightened interest in biosensing and the need to conduct sensitive bio-assays in liquid media served as the catalyst for the development of new classes of acoustic plate mode (APM) devices that do operate in liquids and afford very high sensitivity to surface mass changes.

While these acoustic wave-based sensors are intrinsically mass-producible and inexpensive in high volume production, they have not yet been employed in any high volume analytical sensor applications. Instead, they have been most successful when used in specialized instruments where they offer some uniquely superior performance characteristic. As these technologies mature, one can anticipate the introduction of a diverse array of new, high performance, field portable instrumentation that is based on piezoelectric microsensor technology.

One objective of this presentation is to briefly describe several state-of-the-art instruments that are well suited for the problem of field screening and which use piezoelectric SAW vapor microsensors as their core technology. These include a hand-held instrument for detecting trace quantities of chemical warfare agent vapors, and a unique hand-held instrument that can rapidly learn and identify vapors based on their SAW microsensor response signatures.

References: (1) G. Sauerbrey, Verwendung von Schwingquarzen zur Wagung dunner Schicten und zur Mikrowagung, Z. Phys. 155 (1959)206-222.

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HYPHENATED TECHNIQUES; THE NEXT GENERATION OF FIELD PORTABLE ANALYTICAL INSTRUMENTS?

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Direct, coupling of compatible analytical techniques in tandem, also known as "hyphenation" e.g., GC/MS, MS/MS (or even MS^n), GC/IMS and GC/GC, has the potential of providing a dramatic increase in specificity. Under favorable conditions, this may be accompanied by increased speed (due to reduced sample preparation time) as well as lower detection limits (due to reduced background interference. The information content of the 2-dimensional or n-dimensional data obtained can be several orders of magnitude higher than that of a single spectrum or chromatogram. This distinguishes hyphenated (i.e., serially coupled) methods from parallel coupled dual column and/or dual detector techniques which are also gaining popularity in field analytical applications.

The best known tandem method, GC/MS, arguably the most powerful analytical method for complex mixtures of organic compounds with measurable vapor pressures, has all but replaced the use of one-dimensional GC and MS techniques in the laboratory. In fact, the use of standardized GC/MS procedures has become mandatory for numerous environmental, occupational, clinical, pharmacological and forensic tests, especially if the results may need to stand up in court. Currently, few analytical chemists would dare to propose the use of one-dimensional GC or MS techniques to characterize complex environmental samples. Yet, this is precisely the current state-of-the-art in field portable analytical techniques.

Only a few years ago, it would have seemed preposterous to propose the development of man-portable - let alone hand-portable - tandem instruments in view of the notoriously high complexity, weight and cost of such equipment. However, fully functional prototypes of man-portable GC/MS and hand-portable GC/IMS (gas chromatography/ion mobility spectrometry) systems have already been developed in our laboratory as well as by several other groups (as reported at the previous Las Vegas meeting) and the enticing prospect of personalized, pocket-sized GC/IMS or GC/GC systems has been held forward by some researchers. However, before the birth of the "tri-corder" (Star Trek) can be announced, major hurdles remain to be overcome, particularly in the area of intelligent, man-machine interfaces including data reduction and visualization techniques, self-adaptive learning and pattern recognition methods and, last but not least, advanced telemetry technology.

APPLICATION OF LUMINESCENCE TO FIELD SCREENING OF CHEMICAL CONTAMINANTS IN THE ENVIRONMENT

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Molecular fluorescence and phosphorescence can be measured at very low concentrations for many important classes of organic contaminants. Because luminescence measurements are inherently more sensitive than other spectroscopies, such as UV/Vis, Raman, infrared (IR), and nuclear magnetic resonance (NMR), they are attractive for application to environmental monitoring. Luminescence techniques have been employed as analytical methods for a variety of environmental contaminants. The use of synchronous scanning and fluorescence lifetime filtering and matrix effects can further enhance sensitivity of fluorescence and phosphorescence based methods. The development of fiber-optic based instrumentation should extend the use of luminescence measurements to remote sensing applications. The recent analytical literature on the application of luminescence techniques to analytical measurements of various classes of environmentally significant chemicals has been reviewed. The advantages and disadvantages of luminescence for field screening measurements are discussed. The major advantages projected for luminescence versus alternative instruments, kits, or methods are greater sample throughput, simplicity, greater selectivity and sensitivity, and ease of operation. However, all of these advantages do not apply over every competing technology. The chemical compounds that might be analyzed by luminescence spectroscopy can be classified as follows: compounds that are intrinsically fluorescent, compounds that can form fluorescent derivatives, and compounds that can modulate fluorescence of another compound. Applications of luminescence spectroscopy to all three classes can be found in the literature. Examples include: volatile aromatics, including benzene, toluene, ethylbenzene, and xylene; PCBs, including the Aroclors™; PAHs; phthalates; phenols; some pesticides; several main group elements, such as selenium, tin, and aluminum; actinides and lanthanides; ammonia and various other amines; mercaptans and sulfides. For some of these compounds, other well established methods exist. However, field applications, utilizing a luminescence approach, could serve as a useful adjunct to an existing method. Although the luminescence methods may not always represent the "best" technology, they remain sensitive, fast, versatile, and capable of screening for multiple analytes.

PERFORMANCE SPECIFICATIONS FOR TECHNOLOGY DEVELOPMENT

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The objective of any instrument, technique, or method development project is to *deliver* needed and usable technologies to our customers. To help DOE's Office of Environmental Management (EM) and its investigators meet that goal, technology performance specifications (PS) are being developed for implementation in R&D and DT&E projects. Technology performance specifications will be used to establish milestones, evaluate the status of ongoing projects, and determine the success of completed projects. Preliminary performance specifications will be required in proposals and will be highly weighted in the technical evaluation.

The general performance specification approach is to *document* what currently exists or is nearing completion and *compare* that baseline to the customers' needs to identify the *unmet requirements*. These unmet requirements then form the basis for the technology development needs which OTD investigators must address. The process needs to be *quantitative* where appropriate to focus project goals away from vague generalities like "better" toward specifics like "reduce detection limit from 50 μg/L to 100 ng/L" or "cheaper" to "reduction of labor costs for Step A from 4 hr to 0.5 hr."

A report, Performance Specifications for Technology Development: Application to VOC Characterization and Monitoring in the Environment by SC Carpenter, PV Doskey, MD Erickson, and PC Lindahl, describes the PS model in detail. In addition, the report reviews the application of the available technologies for VOC characterization and monitoring and assesses their applicability to fixed-based labs, mobile labs, transportable, portable, and in-situ measurements.

The application of performance specifications to field analysis for VOCs and other areas will be discussed. Status of implementation within EM will be presented.

FIELD ASSESSMENT SCREENING TEAM

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The Department of Defense, the Department of Energy, and several other federal agencies have long recognized that the cost (and, to a degree, the quality) of environmental site investigations has been disproportionate to the results. The Hazardous Waste Remedial Actions Program (HAZWRAP), as a field organization, initiated a process development program termed Field Assessment Screening Team (FAST) that had as its primary objective the reduction of site characterization costs while maintaining, if not improving, sample quality.

FAST is the technical integration of optimized investigative technology coordinated with on-site, full-suite, site-specific analytical capability and capacity, which is coupled with real-time data management/presentation on an iterative basis. The economic results of this process have been site characterization cost reductions up to 75%, a 50% reduction in field time, and the maintenance of improved data quality.

FAST has been shown to be singularly applicable to the Interim Final Data Quality Objective Process for Superfund as recently issued by the Environmental Protection Agency.

Site characterization costs and the attendant functions represent up to 40% of the total environmental remediation costs. The FAST process could potentially reduce these costs to less than 20% of the total.

THE SAVANNAH RIVER ENVIRONMENTAL TECHNOLOGY FIELD TEST PLATFORM: PHASE II

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The principal goal in the development of new technologies for environmental monitoring and characterization is transferring them to organizations and individuals for use in site assessment and compliance monitoring. The DOE complex has devised several strategies to facilitate this transfer including joint research projects between private industries and government laboratories or universities (CRADAs), and streamlined licensing procedures. One strategy that has been under-utilized is a planned sequence gradually moving from laboratory development and field demonstration to long term evaluation and onsite use. Industrial partnership and commercial production can be initiated at any step based on the performance, market, user needs, and costs associated with the technology. This approach allows use of the technology by onsite groups for compliance monitoring tasks (e.g. Environmental Restoration and Waste Management), while allowing parallel research and development organizations the opportunity to evaluate the long term performance and to make modifications or improvements to the technology. This probationary period also provides regulatory organizations, potential industrial partners, and potential users with the opportunity to evaluate the technology's performance and its utility for implementation in environmental characterization and monitoring programs.

The Savannah River Technology Center (SRTC) has been developing a program to rigorously field test promising environmental technologies that have not undergone EPA equivalency testing. The infrastructure and staff expertise developed as part of the activities of the Savannah River Integrated Demonstration Program (i.e., wells, available power, conventional baseline characterization and monitoring equipment, shelter structures) allows field testing of technologies without the difficulties of providing remote field support. By providing a well-characterized site and a well-developed infrastructure, technologies can be tested for long periods of time to determine their appropriate applications in environmental characterization and monitoring activities. Situation specific evaluations of the technology following stringent test plans can be made in comparison with simultaneous baseline methods and historical data. This program is designed to help expedite regulatory approval and technology transfer to manufacturers and the user community.

An advisory committee has been established consisting of representatives from local and national regulatory groups, industry, universities, public interest groups, potential users, and SRTC personnel. The group helps determine the technologies that are evaluated and reviews the evaluation strategy and specific test plans for the new technology. The inclusion of local regulatory personnel in the advisory groups facilitates the use of appropriate innovative technologies in remedial facility investigations and remediation programs at SRS. Successful internal transfers of technologies at SRS from research and development to users (e.g. Environmental Restoration, Industrial Health and Hygiene) include: 1) gas chromatography using the headspace method to rapidly evaluate volatile organic contamination in water and soil samples; 2) a commercial infrared photoacoustic gas analyzer for faster and more accurate measurements; and 3) the cone penetrometer with several sensors and samplers for rapid, depth discrete contaminant measurements. These internal transfers have stimulated the use of the technologies in both onsite and offsite activities. The cone penetrometer and the headspace method are now accepted field screening techniques in most areas of the country.

As the new technologies are used, there is a larger demand for their implementation both on and offsite. This demand leads industry and users to the technologies. Successful implementation of a technology as compared with baseline technologies should facilitate their acceptance as standard protocols.

RESPONSE TO GASOLINE AND DIESEL CONTAMINATED GROUNDWATER

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ChemSensor is a rugged, field-portable instrument capable of in-situ measurement of organic compounds, especially volatile organic compounds (VOCs) in water and process streams. The response to organics is reversible, requiring only that the user clean and re-zero the probe between measurements. ChemSensor gives field sampling personnel an indication of the concentration at a site in real time. This helps in the assessment of leaking underground storage tank (UST) sites prior to receipt of analytical lab test results and also provides a tool for monitoring the cleanup at a site over time. This paper discusses how to interpret the results obtained from ChemSensor for site assessment at both gasoline and diesel contaminated UST sites.

ChemSensor consists of a probe connected to a meter via electrical cable that enables in-situ sampling to standard depths up to 100 feet (custom lengths longer than 100 feet also available). The sensing element of the probe is a quartz optical fiber coated with a hydrophobic and organophyllic chemical coating sensitive to organic compounds. ChemSensor responds to virtually all organic species dissolved in water, dependent upon the aqueous solubility and refractive index of the compound.

Samples were obtained from both gasoline and diesel fuel contaminated sites and analyzed with ChemSensor and in the laboratory using a gas chromatograph. A linear relationship between ChemSensor and the gas chromatograph data was demonstrated for total BTEX (gasoline) and total petroleum hydrocarbons (diesel fuel). A discussion of the interpretation of ChemSensor data for monitoring contamination levels and remediation processes is presented.

The test results demonstrate that ChemSensor is a useful field portable analytical tool for determining concentrations of total organic compounds in water and a timely supplement to laboratory results. Interpretation of the data from ChemSensor monitoring of a remediation site over time can give the user an indication of the progress of the clean-up without requiring excessive characterization of the response via confirmatory laboratory analysis. ChemSensor can also be used to optimize the collection of samples for laboratory analysis at a new site by indicating the level of contamination within and among the wells in real time.

DIELECTRIC SOIL PROBING TOOL TO MONITOR THE SUBSURFACE DISTRIBUTION OF NON-AQUEOUS PHASE LIQUIDS

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A unique dielectric soil probing tool to monitor the subsurface distribution of non-aqueous phase liquids (NAPLs) is developed. Field tests were carried out down to 30 meters, to find waste layers (NAPLs). The results are interesting and useful. The probe is low cost and simple to use. It is able to give routinely a measure of the soil type (sand, silt, clay), the ion concentration, the water content and the electrical conductivity of soil layers. It is also able to locate the position of DNAPLs and LNAPLs. To make the probe robust and low cost, we designed an Application Specific Integrated Circuit (ASIC) for the high frequency front-end signal processing. The ASIC can be mounted inside the probe close to the electrodes. The output is RS232 for easy connection to some kind of computer or recorder.

Though already useful, only the basic idea has been tested now. There are, however, many new features in development for this technique, to which some already are working in the laboratory. The probe is also useful for continuous.monitoring.

DEVELOPMENT OF AN INFRARED FIBER OPTIC SENSOR FOR IDENTIFICATION OF HAZARDOUS WASTES

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Normal operations of the Air Force generate significant quantities of waste organic materials that must be disposed of in an environmentally acceptable manner. These hazardous liquid wastes include varying quantities of fuel, acids, paints, thinners, strippers, solvents and oils. These wastes are collected in the various shops and then delivered to centralized collection sites at the base where they are typically stored temporarily in 55 gallon drums or other suitable tanks pending delivery to a disposal facility. During this collection process it is not uncommon for the liquid wastes gathered from different shops to be combined in a single storage tank or drum. This creates a complex mixture containing a variety of different organic materials in unknown concentrations.

This poster presentation summarizes the effort to demonstrate the feasibility of developing a field-portable instrument that can perform a quick and accurate chemical analysis of unknown waste materials at Air Force bases. The development of this instrument is required in order to expedite and reduce the cost of hazardous liquid waste disposal. This instrument can perform the analysis without removing a sample from the storage container. The feasibility study demonstrated that devices containing an infrared fiber optic sensor can detect and quantify the entire range of liquid hazardous waste typically found at Air Force bases. The analytical technique that was used is Fourier Transform Infrared Spectroscopy (FTIR). The demonstration involved the fabrication of three different configurations of sensing devices, each of which was shown to accurately predict mixtures of solvents. The overall objective of the current development effort is to deliver to the Air Force a system that will enable Air Force personnel to perform on-site analysis/screening of liquid wastes typically found on bases without removing the sample from the storage container.

CONE PENETROMETER-MOUNTED CHEMICAL SENSOR DEMONSTRATION

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Developments in the fields of geophysics, analytical spectroscopy, and foundation engineering are being brought together to create a powerful tool for hazardous waste characterization. Originally developed to provide engineering data, the cone penetrometer (CPT) has become a vehicle for adaptations of geophysical and spectroscopic techniques. With some distinct advantages over traditional drilling methods for hazardous waste work, there is need and interest in producing information on the advantages and limitations of cone penetrometers in general. More specifically, with the rapid development or adaptation of sensors to the cone penetrometers, there is also tremendous interest in a thorough evaluation of these sensors under real site conditions.

Under the EPA's Superfund Innovative Technology Evaluation (SITE) program, the Monitoring and Measurement Technology Program (MMTP) is charged with demonstrating and evaluating monitoring and measurement technologies. In response to input of EPA Regional Office needs, a demonstration of cone penetrometer-mounted chemical sensors was conducted in August and September of 1994. This demonstration evaluated the spectrum of available cone penetrometer sensor technology. Representing the smaller sized units was Geoprobe Systems, while new optical analytical spectroscopy sensors were represented by the Rapid Optical Analytical Tool, or ROST, (a commercial system) developed by Unysis Corporation and the U.S. Army's Site Characterization and Analysis Penetrometer System (SCAPS) In addition, standard commercially-available, full-sized sensors were evaluated from a contracted rig made available to the demonstration participants by the MMTP Traditional data for comparison were obtained by contracted drill rig and off-site analytical services.

For the purpose of this demonstration, three sites were used including the York Gas and Electric Company Coal Gasification Site in York, Nebraska, which has polyaromtic hydrocarbons (PAH) contamination; a former underground storage tank (UST) site at Ft. Riley, Kansas which has contamination resulting from fuel leaks; and the Atlantic Coal Gas Company site located in Atlantic, Iowa which has both PAH and fuel contamination. The various technologies and the drill rig independently examined through the three sites during the period 13 August to 2 September, 1994.

The demonstration sought to evaluate the participating systems in three main areas including: (1) how the cone penetrometer systems data compared with conventional analytical results (pollution detection), and (2) their ability to provide vertical profiles of the contamination (pollution characterization), and (3) how well they can characterize and sample the subsurface materials (geotechnical characterization) Additionally, all MMTP demonstrations seek to evaluate such planning factors as reliability, ruggedness, cost, operating range, required QA/QC parameters, training requirements, and ease of operation as compared to conventional techniques.

As the demonstration is only recently concluded and the data has not yet been completely evaluated, this discussion seeks to provide basic information about the participating technologies and the conduct of the field activities. The final report of the demonstration is scheduled to be completed in early 1995

Notice; This abstract was prepared for an oral presentation. It does not necessarily reflect the views of the U S EPA. Mention of trade names does not constitute endorsement or recommendation for use.

POSSIBILITIES FOR APPLICATION OF FIELD SCREENING METHODS WITH SENSOR TECHNIQUE IN ENVIRONMENTAL ANALYSIS

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The formerly used methods of taking samples and carrying out lab analysis for soil, waste and toxic deposits require a lot of time and money. Because of this, usually only a small amount of samples is analyzed. The few samples and the complex environmental matrices are the reason why the results of analysis vary so much and are difficult to discuss. The intention of the study "Possibilities of application of Field Screening Methods with Sensor technique" , which was started in 1992, was to investigate the market to find new methods and instruments for detection of toxic compounds in the area of environmental protection. With sensor technique analysis should get faster and cheaper. Another purpose of the study is to raise the acceptance of Field Screening Methods at the responsible agencies.

First the national and international literature was investigated for new methods and instruments. The result was a list of about 20 instruments which looked interesting and were chosen to be tested under field conditions. Some selected results of the first part of these field tests are documented in this poster. The tests were realized on the site of a former sawmill with wood impregnation in Sinsheim, Germany, where the pollutants PAH, aliphatic Hydrocarbons, PCP and Chrome were found.

The following methods and instruments were tested, compared and evaluated up to now:

* Immunoassays (PAH, aliphatic Hydrocarbons, PCP)
* Mobile GC/MS (organic compounds)
* Mobile XRF (HM)
* Lasersonde (PAH)
* Mobile IR-instruments (aliphatic and aromatic Hydrocarbons)
* Luminescent bacteria test

COMPARISON OF DIRECT SAMPLING ION TRAP MASS SPECTROMETRY TO GC/MS FOR MONITORING VOCS IN GROUNDWATER

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Rapid analytical methods which utilize Direct Sampling Ion Trap Mass Spectrometry for field screening and compliance monitoring of volatile organic compounds in groundwater have been under development in our laboratory for several years. These methods can be used on fieldable instrumentation, have low ppb detection limits, and require little or no sample preparation. The DSITMS analysis of VOCs in water uses a three minute direct purge of the water sample with a stream of helium at a flow rate of 100 mL/min. VOCs partition into the helium stream and are carried directly into the ion trap via a 100 um section of fused silica capillary. Most of the sample is vented through an open/split interface, with only about 1 mL/min. entering the mass spectrometer. Analysis of the VOCs is accomplished by alternating conditions in the ion trap at roughly one second intervals between electron ionization and chemical ionization (water reagent gas). Two such methods for the analysis of VOCs in water have been submitted to EPA for regulatory approval, and in order to facilitate this process, validation studies have been conducted comparing the DSITMS method to a standard method such as purge-and-trap GC/MS. One such study, recently completed, involved the analysis of over 100 groundwater samples collected from the Oak Ridge reservation for compliance monitoring. Split samples of the groundwater were analyzed by DSITMS and by purge-and-trap GC/MS at a local CLP laboratory. The results of the comparative study generally show excellent qualitative and quantitative agreement between the DSITMS and standard methods, with the DSITMS method being superior with respect to speed, cost, ease of use, and minimized artifacts.

THERMAL SOIL DESORPTION FOR TOTAL PETROLEUM HYDROCARBON TESTING ON GAS CHROMATOGRAPH

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Testing for *total petroleum hydrocarbons* (TPH) is one of the most common analytical tests today. A recent development in chromatography incorporates *Thermal Soil Desorption* technology to enable analyses of unprepared soil samples for volatiles such as BTEX components and semi-volatiles such as diesel, PCBs, PAHs and pesticides in the same chromatogram, while in the field.

A gas chromatograph is the preferred method for determining TPH because the column in a GC separates the individual hydrocarbons compounds such as benzene and toluene from each other and measures each individually. A GC analysis will determine not only the total amount of hydrocarbon, but also whether it is gasoline, diesel or another compound.

TPH analysis with a GC is typically conducted with a Flame Ionization Detector (FID). Extensive field and laboratory testing has shown that incorporation of a Thermal Soil Desorber offers many benefits over traditional analytical testing methods such as Headspace, Solvent Extraction, and Purge & Trap.

This paper presents the process of implementing Thermal Soil Desorption in gas chromatography, including procedures for, and advantages of faster testing and analysis times, concurrent volatile and semi-volatile analysis, minimized sample manipulation, single gas (H_2) operation, and detection to the part-per billion levels.

ON-SITE MEASUREMENTS
A GUIDANCE DOCUMENT FOR DECISIONS USING ON-SITE ANALYTICAL SUPPORT

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The purpose of this breakout session will be to discuss the direction of the draft document, On-Site Measurements - A Guidance Document for Decisions Using On-Site Analytical Support" and to provide input to the developers for improvement and to make it useful across Government and non-Government organizations. This document was developed under the auspices of DOE's Analytical Services Division (EM-263) to bring legitimacy to the appropriate use of on-site measurements and to provide a rationale to the DOE complex decision makers for using on-site measurements (real time and pseudo-real time) for making necessary field decisions.

The authors believe that there are three potential reasons for using on-site measurements (1) in situ monitoring/measurements, (2) provision of data for decisions that must be made in the field in real time, and (3) an option that falls out of the DQO process step 7, "optimization of design," that is the most cost effective design for meeting project goals. The document under discussion focusses on reason number 2.

Examples of using on-situ measurements to make decisions in real time are (1) to redirect sampling efforts based on new knowledge; (2) to determine i an area of interest, such as a project-specific "exposure unit" (EU) is clean or needs to be remediated, based on (e.g.) sampling sub-plots and analyzing composites that represent the EU while the removal equipment is still there, and (3) to determine the best locations for monitoring wells.

The document describes program elements needed for success such as how to incorporate the use of on-site measurements for decision making into the planning process to assure that the data quality is adequate for making those decisions the kinds of QA and QC issues that must be addressed to support the use of the on-site measurements and to document the quality, and how to determine the adequacy of the data after it is collected to justify its use.